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| 13. ABSTRACT (Maximum 200 Words) The SCAPS XRF Metals Sensor adds metals detection capability to the DOD Site Characterization Penetrometer System (SCAPS). It works using X-ray Fluorescence (XRF) to detect all metals above calcium in the subsurface soil. The sensor head contains a miniature x-ray tube and cooled solid-state detector to achieve performance comparable to laboratory units in a down-hole sensor. The sensor may be pushed to depths as far as 150 feet by the cone penetrometer system and is designed to detect metals at concentrations below 100 ppm in all soil types regardless of moisture level. The system has been tested on the bench and demonstrated at a site. Following a brief overview of the sensor design and operation, the results of the bench testing, including evaluation of detection limits for a list of common contaminant metals, will be presented. Some operational test results are included, both on technical performance of the sensor and its robustness and effectiveness as a field tool. Finally, a list of potential improvements is given. | | | | |
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Field Demonstration of the SCAPS XRF Metals Sensor

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ABSTRACT

The SCAPS XRF Metals Sensor adds metals detection capability to the DOD Site Characterization Penetrometer System (SCAPS). It works using X-ray Fluorescence (XRF) to detect all metals above calcium in the subsurface soil. The sensor head contains a miniature x-ray tube and cooled solid-state detector to achieve performance comparable to laboratory units in a down-hole sensor. The sensor may be pushed to depths as far as 150 feet by the cone penetrometer system and is designed to detect metals at concentrations below 100 ppm in all soil types regardless of moisture level.

The system has been tested on the bench and demonstrated at a site. Following a brief overview of the sensor design and operation, the results of the bench testing, including evaluation of detection limits for a list of common contaminant metals, will be presented. Some operational test results are included, both on technical performance of the sensor and its robustness and effectiveness as a field tool. Finally, a list of potential improvements is given.

INTRODUCTION

To be deployed via a cone penetrometer system, a metals sensor must meet several constraints. First and foremost, it must fit within the constraints of the penetrometer pipe. Even the larger push pipes used for sensors have a maximum inside diameter of 1-3/4 inch. This places severe constraints on the layout and configuration of the components of the sensor. Second, it must be rugged enough to withstand being pushed (as well as the rigors of typical field use). Finally, each sensor must have an x-ray transparent window to access the soil which is strong and hard enough to withstand the soil abrasion and pressure and yet be transparent to the sensor radiation. A metals sensor should be able to detect as wide a variety of metals as possible and should have detection limits well below 100 ppm for field screening. Finally, to operate efficiently during typical penetrometer operation the sensor should be able to collect data in about 100 seconds or less. Such speed is necessary to accomplish three-dimensional mapping of a reasonable area in a few days. Techniques that require any type of sample processing (especially wet chemistry) or that the sample to be placed in a cell for transmission measurements are extremely difficult at best.

To achieve the goals, a sensor was constructed and tested for the DOD Tri-Service SCAPS program (Site Characterization and Analysis Penetrometer System) using x-ray fluorescence (XRF) as the metals detection mechanism. XRF requires no sample preparation and is nondestructive. The typical geometry uses an excitation beam and a

detector, each at 45 degrees to the sample. The spectrum is relatively easy to interpret, containing typically two peaks per element with few ambiguities and overlaps. All elements with atomic number above calcium can be detected, with detection limits of a few ppm in the laboratory. Data reduction is straightforward, with matrix effects and calibration understood well enough to easily achieve the accuracy required for field screening.

DESCRIPTION OF SENSOR

The XRF metals sensor consists of three subsystems: the actual below-ground probe, the umbilical cable, and the above-ground electronics package. The probe contains the x-ray source, detector and preamp, appropriate x-ray optics, the mounting system, and the rugged x-ray window. A sealed x-ray tube is used as the source both in order to achieve adequate detection limits in the allotted time and to avoid the licensing and safety problems of a radioactive source. Since probes are occasionally lost at depth, a radioactive source poses unacceptable risks. The detector is a silicon PIN diode in a small case with self-contained cooling, connected to a low-noise preamp in close proximity. The preamp provides sufficient signal to drive the umbilical cable. The x-ray window is boron carbide, the second hardest material currently available. Since it consists of low atomic number materials it is relatively transparent to x-rays in the relevant energy range.

The umbilical cable conducts the high voltage and filament power required by the x-ray tube, the electronics and cooling power for the detector and preamp, and the signal pulses from the detector. It is fully shielded both for noise immunity and high voltage safety. It also contains the signal cables required by the usual penetrometer stratigraphy sensors.

The above ground electronics package contains the electronics necessary for the x-ray tube and for the detector. The x-ray power supply for the x-ray tube provides high voltage (30kV) and filament voltage, which is regulated to provide constant emission current and thus constant x-ray output. The detector electronics provides the necessary power supplies and contains the pulse-shaping circuitry for the detector. Its output connects to a standard multichannel analyzer for data collection and analysis. The electronics package connects to an interlock shield which insures that the probe is inserted into the ground before the x-ray tube is energized. This shield also allows test samples to be measured above ground via a sample introduction port. The remainder of the above-ground system consists of a multichannel analyzer and a portable computer to collect, interpret, and display the data. Software is available to decode the spectrum and provide a list of elements present as well as an interpretation of the amount in parts per million (by weight).

Commercial components were used to the greatest extent possible. The x-ray tube was custom fabricated by a commercial manufacturer to our specifications. The only major component made in-house was the x-ray power supply, which must have a split design with a down-hole filament isolation transformer.

The x-ray optical design follows the feasibility tests performed earlier¹. A molybdenum anode is used to excite the majority of contaminant metals. An aperture allows only the soil window to be irradiated by the x-ray beam. The beam passes through a molybdenum filter to attenuate the continuum radiation and reduce background (to improve the detection limits).

The mechanical design employs a rail for mounting the components, assuring rigidity, ruggedness, and accurate pre-alignment on the bench. This also provides compatibility with other sensors and housing designs. A central collimator block mounts the x-ray tube

and provides collimation of the beam and a mount for the filter. This collimator also positions the detector and contains a collimator for the detector. This integral collimator block insures proper relative alignment of the x-ray tube and detector. Slots in the mounting rail allow the collimator block to be aligned with the window.

The layout of the sensor is shown in Figure 1 below.

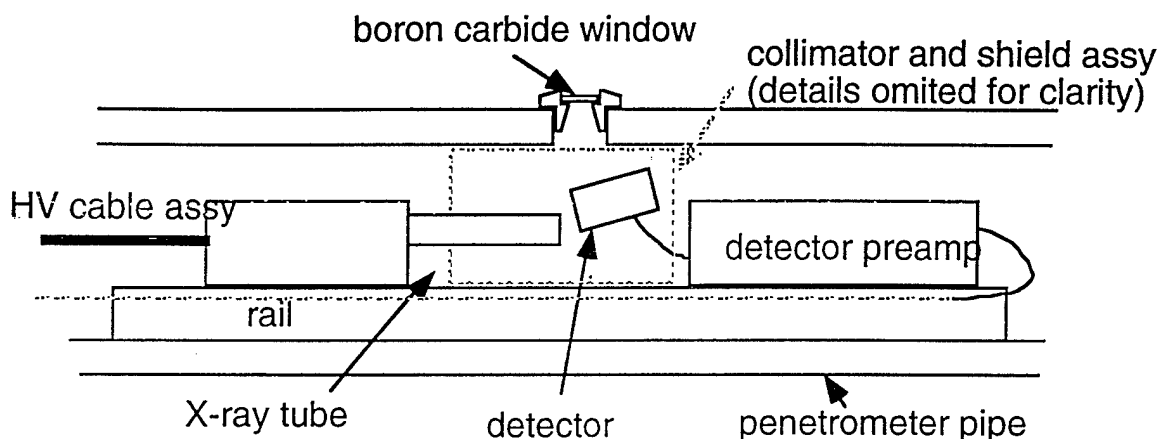


Figure 1. Engineering layout of the SCAPS XRF Metals Sensor

SENSOR BENCH TESTS

At present the sensor has been successfully constructed and is undergoing evaluation and field demonstration. Spectra were taken on the bench to determine detection limits. An underground test during an actual penetrometer push was also performed to verify the operation of the probe under actual usage conditions.

The bench tests were performed with the sensor lying on its side in a radiation enclosure and the samples placed on top of the window in plastic sample cups. Two of the samples were National Institute of Standards and Technology Standard Reference Materials, SRM 2710 and SRM 2711. These are Montana soils with elevated amounts of heavy metal contaminants which have been carefully characterized. These reference standards contain both lead and zinc at levels between several hundred and several thousand ppm. In addition, samples consisting of metal salts mixed with cellulose, dried, and pressed into discs were measured to determine detection limits for metals not in the NIST samples. The cellulose discs contained copper and mercury at concentrations from 1000 to over 10,000 ppm.

A spectrum taken with the sensor on the bench is shown in Figure 2. The sample is SRM 2710 and the iron, zinc, and lead peaks can be easily seen. The zinc and lead are present at levels near 6000 ppm. In addition, the peak for copper can be seen just to the left of the zinc peak, and the iron and zinc K beta peaks can be seen just to the right of the main (K alpha) peaks for these elements. The large peak to the right is scatter of the incident spectrum from the x-ray tube by the window and the soil. The spectrum was collected in 100 seconds, just as it would be in a typical site screening operation.

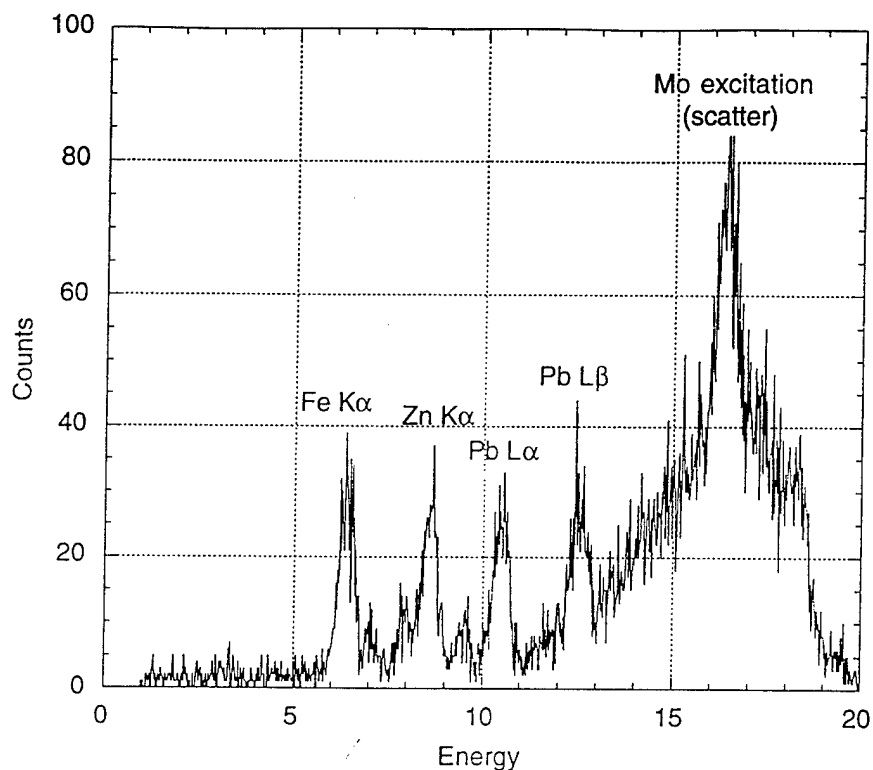


Figure 2. Spectrum taken in 100 seconds with the SCAPS XRF Metals Sensor. Note the contaminant peaks for zinc and lead. The sample is SRM2710, Montana soil with highly elevated heavy metal contaminants (about 6000 ppm for zinc and lead).

The detection limits for a few common contaminants are listed in table I. These numbers represent averages of several measurements, both repeats as well as measurements with different concentrations of the metal of interest.

Table I. Detection limits for 100 second counting times with the operational configuration of the SCAPS XRF Metals Sensor.

| Metal | 3 s Detection Limits | without window |
|---------|----------------------|----------------|
| Lead | 326 | 136 |
| Mercury | 254 | 106 |
| Copper | 88 | |
| Zinc | 214 | |

The limits are three-sigma limits, determined via the formula in Equation 1.

$$D. L. = \frac{3\sqrt{bkg}}{int} \times conc \quad (1)$$

where

int = integrated intensity of the peak from the metal of interest
 bkg = background integral under the peak
 conc = concentration of the metal in the sample in ppm (by weight).

The limits other than copper are somewhat higher than the desired 100 ppm, but still below the limits for hazardous waste for most heavy metals except mercury. These limits represent those obtainable with 100 second counting times; longer times would yield lower limits. The principal detriment to the limits is the scatter by the x-ray window. To provide some quantitative measure of its effect, the window was removed and the detection limits for both lead and mercury re-evaluated. These numbers are also shown in Table I. As can be seen, the limits are better by almost a factor of three without the window. The window used here is of a very conservative design² to insure survival of the probe during initial field tests. Experiments with window ruggedness indicate a thinner window could be used, which would significantly improve detection limits. Other factors under investigation to improve detection limits are the use of a larger window diameter and higher x-ray tube power. The tube was operated at a voltage of 30 kV and a current of 30 microamperes, for a power of 1 watt. Future increases in both the voltage and current will directly improve detection limits.

UNDERGROUND TEST

To verify the operation of the sensor in its intended usage, a test was performed with the sensor pushed by a penetrometer to depths up to six feet. The sensor was exercised during the entire operation to verify its ruggedness and ability to operate during a push. The sensor was able to collect data during all phases of its deployment, including descent, ascent, while stationary, and measuring samples in the truck.

The only problem encountered was that the x-ray detector would cease to count for periods of about 100 seconds at random times. This behavior began sometime during the underground part of the test and persisted after the sensor was returned to the laboratory. Shortly after this, the detector returned to normal and gave no additional trouble. Subsequent tests in the laboratory showed only a tendency to microphonics when the sensor was delivered a sharp blow, and no effects of mishandling otherwise. The microphonics are easily observed in the spectrum, and no evidence for them was found during penetrometer operations.

The area where the underground test was performed was a vacant lot at the USAE Waterways Experiment Station in Vicksburg, Mississippi. Since the soil contains few if any contaminants at this spot, only the naturally occurring iron peak was observed initially. To demonstrate proper operation of the sensor, a few brass shavings were carefully placed in a six foot hole and the probe pushed until the sensor window was adjacent to the shavings. The spectrum collected at this point is shown in Figure 3. The brass peaks are very large, indicating that one of the shavings was against the window. This spectrum was collected for a considerable time to achieve good counting statistics and a smooth curve for display. Note the presence of the minority lead peaks in the brass.

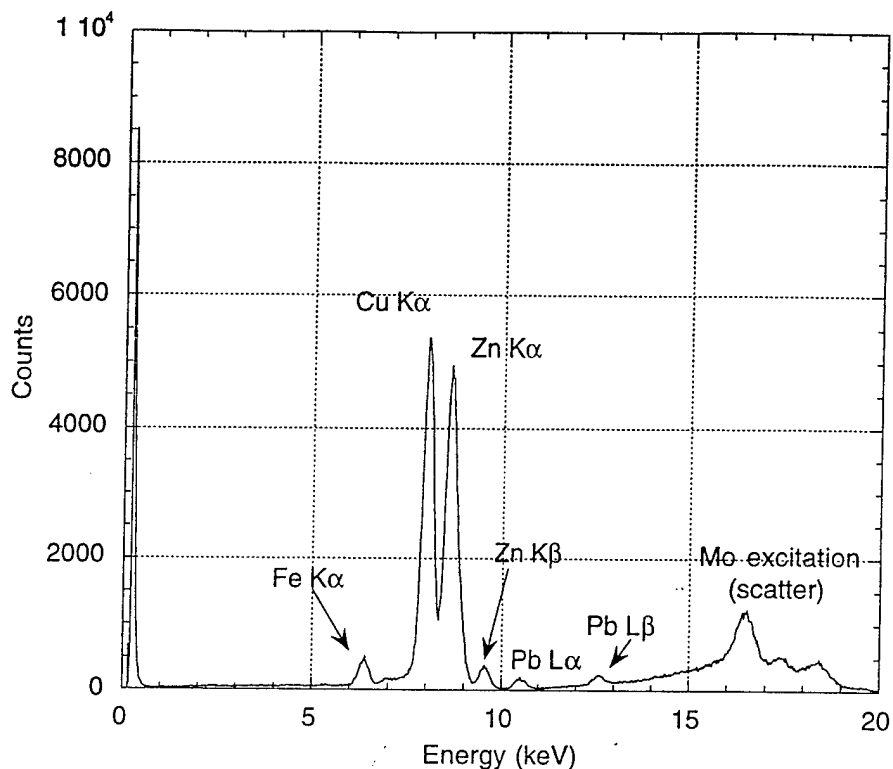


Figure 3. Spectrum of brass shavings in a six foot hole at the USAE Waterways Experiment Station. A long counting time was used to produce a smooth spectrum for display. Note the detection of the minority lead in the brass.

FUTURE PLANS

The next step is to test the sensor at an actual contaminated site to determine its performance under typical field conditions and in the application for which it was intended. This test is currently scheduled for March 1997, with the exact site to be determined.

The sensor is designed to mate with the usual soil classification tip employed by the penetrometer system. The umbilical contains all of the required leads and provision is made for passing them through the metals sensor. Not only will this allow simultaneous stratigraphy information to be obtained, but the soil classification can be used to help quantify the XRF results for specific metals at the site under investigation.

Several improvements to the current design are being considered. The first of these will be to replace the x-ray window with a thinner window and possibly with a higher purity material. Other window materials, such as diamond films grown by chemical vapor deposition, will be considered. Any replacement window must be tested to insure it will withstand the physical rigors of the penetrometer system before it is used on the sensor.